PROJECT NUMBER: 6505
PROJECT TITLE: Special Investigations/Methods Development
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PERIOD COVERED: June, 1988

I. METHODS FOR CASINGS AND AFTERCUTS

A. **Objective**: To develop rapid methods for the quantitation of propylene glycol, glycerin, ethanol, water, and isosweet in casings and aftercuts.

B. **Results**: Standards containing propylene glycol, glycerin, water, and ethanol in the range of 10-50 mg/mL were analyzed on Porapak® QS and Porapak® PS columns using oven temperature programming to optimize peak resolution. While the peaks are resolved on both columns, the efficiency of the PS column is greater than that of the QS column. Analysis times are about seven and eight minutes for the QS and PS columns, respectively. Response (as peak area) of the standards is linear for all four components on both columns. Dilute solutions of top and bright casings were also analyzed on each column. Interferences observed for the bright casing were minimized by modifying the oven temperature program. No interferences were observed for the top casing.

C. **Plans**: Plans are to determine the durability of these columns and to identify an acceptable internal standard.

II. SPECIFICATIONS AND ANALYSIS OF INCOMING BULK LIQUIDS

A. **Objective**: To evaluate the specifications and analytical testing protocol for bulk shipments of propylene glycol, glycerin, isosweet, and ethanol (SD-4).

B. **Status**: Discussions were held with Quality Assurance personnel from Stockton Street, Cabarrus, Park 500, the BL Plant, Louisville, and the Manufacturing Center regarding the following topics: 1) the subjective and quantitative tests currently performed for acceptance of bulk liquids, 2) the tests which are deemed important, 3) the significance of the associated specifications, 4) the tests desired to ensure the quality of the materials, and 5) the instrumentation available in each of the laboratories. This information is being collated on a spread sheet for evaluation.

C. **Plans**: A discussion with personnel from the Flavor Center has been tentatively planned for the week of June 27th. The information gathered will then be issued to the respective facilities for review and additional comments. Following, a meeting will be held with personnel from all locations to discuss the overall evaluation and to establish a standard testing protocol for these bulk liquids.

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III. PROJECT ART

A. **Objective**: Provide analytical support to project ART.

B. **Results**:

The study of the statistical distribution of the amount of ART extracted nicotine from 25 ART runs has been completed. The data have been reported to ART personnel for statistical analysis.

A study was completed on the distribution of nicotine in ART extracted filler as a function of its position in the extraction vessel. Generally, higher levels of nicotine were observed at the top relative to the bottom of the basket.

A series of experiments were performed which showed that over a range of 6% to 40% OV, the higher the water content of the filler, the shorter the extraction time required to extract the nicotine (methanol/ammonia was the extracting solvent). At 10% OV, a three hour extraction yielded 95% of the nicotine extracted at 20% and 40% OV after a one hour extraction. Above 20% OV, extraction times above one hour did not improve the extraction efficiency.

As part of the unextracted nicotine project, many samples were analyzed for nicotine in cooperation with W. Hempfling. One conclusion from this work is that extraction of ART extracted filler with 1 Normal sodium hydroxide at 90 degrees C for 48 hours resulted in approximately 25% more nicotine than off-line pyrolysis of the same. This work will be continued with analysis of the water-washed, filler residue for nicotine; both by extraction and pyrolysis methods.

IV. ANALYSIS OF RESIDUAL SOLVENTS IN PACKAGING MATERIALS

A. **Objective**: To provide headspace analyses for residual solvents from packaging materials and develop a QA method for the routine analysis of packaging materials.

B. **Results**: A draft of the method for residual solvent analysis has been written and is under review. Currently, submitted samples are being analyzed by project 6505 and QA to ensure consistent results can be obtained.

C. **Plans**: Finish preparation of written method and continue running duplicate samples at both locations until consistent agreement is achieved.

V. RESPONSE TO ANALYTICAL REQUESTS

A. **Objective**: To provide analytical support to R&D and Operations personnel and projects.

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B. **Results**:

Analyses and investigations by the project personnel during the month of June included:

A flavor was analyzed for CF content. No CF was detected.

Eight packs of Marlboro were analyzed for ethanol content as part of Marlboro Standardization. Ethanol values ranged from 69 to 207 µg/cigarette.

Four customer complaint samples were received and analyzed for possible contaminants. The results were reported to QA.

Samples of Chewbacco Nicotine gum were subjected to various analyses as part of an overall attempt to determine the contents of the gum. The average nicotine concentration was 0.45 mg/piece (one piece weighed about 1.0 g) before chewing and 0.010 mg/piece after chewing for one half hour. Metal analysis by XRF showed primarily Ca and Ti. The amount of tobacco material in the gum was estimated to be less than 1% by weight. Determination of glycerin, PG, and triacetin gave 0.27%, 0.05%, and 4.2%, respectively. GC/MS analysis of an extract of the gum showed the balance of the volatile material to be mainly hydrocarbons, plus smaller amounts of acetate esters and other unidentified compounds.

The amount of methoprene delivered to MS smoke was determined on PM Super Lights (Japan) after application at the 5 ppm level. Analysis by GC/MSD-SIM showed the delivery to be 0.22 µg/cigarette analyzed on a Cambridge pad, reflecting a 7.3% MS delivery.

Sixty samples from B. Jenkins' MS/SS evaluation were analyzed for nicotine.

Six tower gas samples from M/C and Cabarrus were analyzed for glycerin and nicotine in order to try to define process differences between the two locations.

C. **References**:

1. Ingraham, D. F., "CC 880513090, Control No. 88032 - Marlboro Lts FTB - Mark Lauley," memo to Cindy Spielberg, May 31, 1988.
2. Ingraham, D. F. and Shelton, J. H., "CC 880505011, Control No. 88029 - Marlboro Lts Menthol 100's HP - Elyse Brook," memo to Cindy Spielberg, June 3, 1988.
3. Ingraham, D. F. and Shelton, J. H., "Control No. 88030 - Cambridge Lts Menthol 100's - D. Hugh Davis," memo to Cindy Spielberg, June 3, 1988.
4. Ingraham, D. F. and Shelton, J. H., "CC 880526011, Control No. 88036 - Marlboro KS - Pamela Thorn," memo to Cindy Spielberg, June 17, 1988.
5. Kanipe, B., "CHEWBACCO Gum - 'Tobacco' Content," memo to B. Handy, June 10, 1988.

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